IS 11135 (1984): Specification for Metuxuron, Technical
[FAD 1: Pesticides and Pesticides Residue Analysis]

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Indian Standard

SPECIFICATION FOR METOXURON, TECHNICAL

UDC 632·954 METOXURON

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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SPECIFICATION FOR METOXURON, TECHNICAL

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Indian Standard

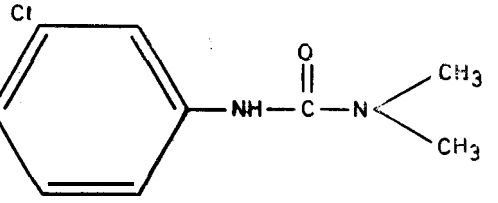
SPECIFICATION FOR METOXURON, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 February 1985, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Metoxuron, technical is employed in the formulation of herbicidal preparations for use on agriculture crops.

0.3 Metoxuron is the accepted common name by the International Organization for Standardization (ISO) for 3-(3-chloro-4 methoxyphenyl) 1, 1-dimethyl urea. The empirical, structural and molecular mass of metoxuron are given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_{10}H_{13}Cl\,N_2O_2$		228·7

0.4 In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act,* 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the

*Rules for rounding off numerical values (*revised*).

rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling line free and test for metoxuron, technical.

2. REQUIREMENTS

2.1 The material shall be in the form of white to off-white colour crystal-flowing powder.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR METOXURON, TECHNICAL

Sl No.	Characteristic	Requirement	Method of Test, Ref To	
			Appendix of this Standard	Clause No. of IS : 6940-1982*
(1)	(2)	(3)	(4)	(5)
i)	Metoxuron content, percent by mass, *Min*	97·0	A	—
ii)	Melting point	120 to 125°C	—	6
iii)	Material insoluble in acetone, percent by mass, *Max*	1·0	—	9
iv)	Water content, percent by mass, *Max*	1'0	—	4

*Methods of test for pesticides and their formulations (*first revision*).

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in clean and dry containers made of mild steel or tin plate or fibre-board or double hessian jute bags (see IS : 8115-1976*) or DW tarpaulin laminated jute bags (see IS : 8117-1976†) or HDPE woven sacks (see IS : 8069-I 976‡). The container shall also comply with the general requirements as stipulated in 2 of IS : 8190 (Part 1)-1980§.

*Specification for double hessian jute bags for pesticides.
†Specification for DW tarpaulin laminated jute bags for pesticides.
‡Specification for high density polyethylene (HDPE) for packing pesticides.
§Requirements for packing of pesticides: Part 1 Solid pesticides (first revision).

3.2 Marking — The container shall bear legibly and indelibly the following information and any other information as is necessary under the *Insecticides Act* and Rules.

a) Name of the material;

b) Name of the manufacturer;

c) Date of manufacture;

d) Batch number;

e) Metoxuron content, percent (*m/m*);

f) Net mass of the contents; and

g) The cautionary notice worded as in *Insecticides Act* and Rules.

3.2.1 The container may also be marked with the IS1 Certification Mark.

NOTE — The use of the IS1 Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks)Act, and the Rules and Regulations made thereunder. The IS1 Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by IS1 and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the IS1 Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of material shall be drawn as prescribed in IS : 10946-1984*.

5. TESTS

5.1 Tests shall be carried out as referred to in col 4 and 5 of Table 1.

5.2 **Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water (see *IS* : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Methods of sampling for technical grade pesticides.

†Specification for water for general laboratory use (second *revision*).

APPENDIX A
[*Table* **1,** *Item* (i)]

DETERMINATION OF METOXURON CONTENT

A-O. GENERAL

A-0.1 Either of the two methods, namely, amine method or total nitrogen method may be used for determining active ingredient content.

A-l. AMINE METHOD

A-l.1 **Apparatus**

A-1.1.1 *Beakers* — 250-ml capacity.

A-1.1.2 *Magnetic Stirrer*

A-1.1.3 *Crucible* — *G-4* porosity.

A-1.1.4 *Filtration Flask* — 500-ml capacity.

A-1.1.5 *Separating Funnel* — 500-ml capacity.

A-1.1.6 *Round Bottom Flask* — 500-ml capacity.

A-l **.1.7** *Rotary Evaporator*

A-1.1.8 Distillation Assembly — as shown in Fig. 1.

A-1.1.9 Glass *Beads*

A-l.2 Reagents

A-1.2.1 *Methylene Dichloride* — pure.

A-1.2.2 *Hydrochloric Acid* — 0·1 *N.*

A-1.2.3 Potassium Hydroxide — pellets.

A-1.2.4 *Boric Acid* — pure.

A-1.2.5 *Mixed Indicator* — *40* mg methylene blue indicator and 60 mg methyl red dissolved in 100-ml alcohol diluted to 250-ml in standard volumetric flask with distilled water.

A-1.2.6 *Propylene Glycol* — pure.

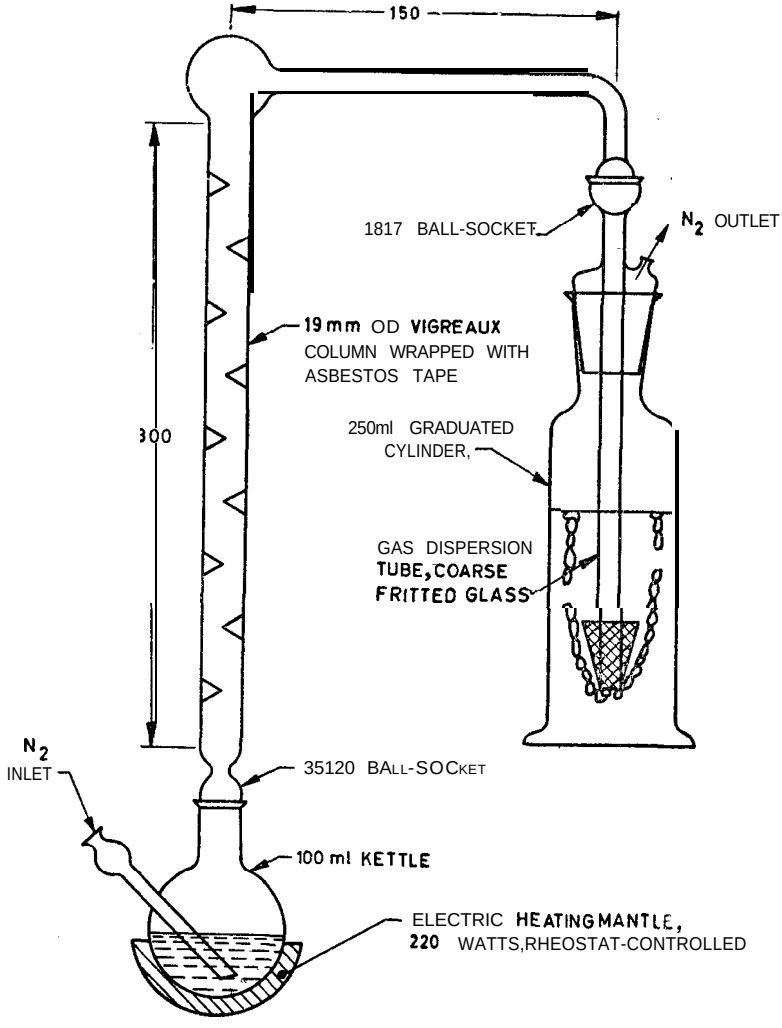


FIG. 1 DISTILLATION ASSEMBLY

A-1.3 Procedure

A-1.3.1 Weigh accurately approximately 3·0 g of sample into a 200-ml beaker. Add 100 ml methylene dichloride and stir by means of a magnetic stirrer for 5 minutes. Filter through fritted glass crucible and rinse the beaker and crucible with portions of methylene dichloride to total volume of approximately 200 ml. Use only slight vacuum to prevent crystallisation of material on walls of crucible. Transfer the quantity to 500-ml separatory funnel and add 50 ml of 1 N hydrochloric acid.

A-1.3.2 Shake the mixture vigorously for one minute and drain the lower organic layer into the second separator. Now add 25 ml of 1 N hydrochloric acid to the organic layer in the second separator and shake vigorously for 30 seconds. Drain the lower layer into a 500-ml round bottom flask. Wash the two acid layers with 100-ml portion of methylene dichloride and drain the lower layer in the 500-ml round bottom flask. Discard the acid.

A-1.3.3 Evaporate methylene dichloride under vacuum in rotary evporator to dryness at a maximum temperature of 40°C. Now add 100 ml propylene glycol and 40 g of potassium hydroxide and some glass beads. Immediately connect flask to distillation apparatus whose joints are lubricated with grease. The end of the condenser delivery tube is dipped below the level of absorbing solution of 0·2 g boric acid, 1 ml mixed indicator in 150 ml water.

A-1.3.4 Gently warm the flask to dissolve all praticles and then boil till propylene glycol distils into condenser. Titrate distilled dimethyl amine continuously with standardized 1 N hydrochloric acid. Complete distillation by slow addition of water (1 drop/second) through a dropping funnel. Continue titration till end point persists for 2 minutes (V ml). Also perform blank (B ml).

A-1.4 Calculation

$$\text{Metoxuron content, percent by mass} = \frac{(V - B) \times N \times 22.87}{M}$$

where

N = normality of hydrochloric acid; and

M = mass, in g, of sample taken for test.

A-2. TOTAL NITROGEN

A-2.1 Apparatus

A-2.1.1 *Kjeldahl Flask* — 500-ml capacity.

A-2.1.2 *Distillation Assembly*

A-2.1.3 *Beakers* — Tall form, 500-ml capacity.

A-2.2 Reagents

A-2.2.1 *Copper Sulphate* — pure.

A-2.2.2 *Potassium Sulphate* — pure.

A-2.2.3 *Sulphuric Acid* — pure.

A-2.2.4 ***Sodium Hydroxide Solution*** — ***40*** percent (m/v).

A-2.2.5 ***Hydrochloric Acid*** — $0{\cdot}5\,N$.

A-2.2.6 Methyl Red Indicator — $0{\cdot}1$ percent in alcohol.

A-2.2.7 ***Sodium Hydroxide Solution*** — $0{\cdot}5\,N$.

A-2.3 Procedure

A-2.3.1 Weigh accurately $1{\cdot}0$ g of sample in a 500-ml Kjeldahlflask and add to it $0{\cdot}5$ g of copper sulphate, 15 g of potassium sulphate and 25 ml of concentrated sulphuric acid. Start heating gently for ten minutes and then strongly till clear or pale green liquid without fumes is obtained. (Approximately 3 to 5 hours.) Transfer the liquid quantitatively to a 1 000-ml round bottom flask by giving washings of about 400-ml distilled water. Add 3 to 4 glass beads and sodium hydroxide solution and start ammonia distillation. The discharge tube should dip below liquid surface into 500-ml beaker covered with a filter paper and containing exactly 50 ml of $0{\cdot}5\,N$ hydrochloric acid. Flow the cooling water into the condenser. Check that the Assembly is airtight. Add 100 ml of 40 percent sodium hydroxide through separating funnel drop by drop. Heat the flask to boiling till evolution of ammonia is complete. Disconnect the apparatus and wash down the condenser with distilled water. Titrate the excess of hydrochloric acid against standard $0{\cdot}5$ Nsodiurn hydroxide (V ml) using phenolphthalein as an indicator. Carry out a reagent blank (B ml).

A-2.3-2 ***Calculation***

$$\text{Metoxuron content, percent by mass} = \frac{(B - V) \times 0{\cdot}5 \times f \times 228{\cdot}7}{M \times 2 \times 10}$$

where

f = factor of $0{\cdot}5$ Nsodium hydroxide used, and

M = mass of sample taken for the test.

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